



CSIRO Land and Water
Environmental Hydrology Group

Radionuclides in the Environment
TRAINING MANUAL
September - November 1999

Prepared by Haralds Alksnis, Danny Hunt and Peter Wallbrink

CSIRO Land and Water
Technical Report 30/99, August 1999
ISBN 0 643 06065 0

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**Main Gamma and Alpha detector room
CSIRO Land and Water
Environmental Hydrology Group**

INTRODUCTION

CSIRO

CSIRO is Australia's premier research body with an annual budget of \$700m and 6000 staff carrying out research in agriculture, environment, manufacturing, minerals and energy, information technology, infrastructure and services. Through its high-quality science and technology, CSIRO provides strategic advice to government, industry, and environment groups.

Land and Water

CSIRO Land and Water's primary role is to carry out and deliver innovative research in soil, water, and related atmospheric processes essential to the sustainable management of land and water in Australia. Land and Water is a multidisciplinary research unit with 480 staff and an annual budget of \$43m. The organisation operates from eight sites across Australia and has a broad range of external sponsors who contribute \$15m to collaborative research. Staff are located in Perth, Adelaide, Albury, Griffith, Canberra, Brisbane and Townsville.

Environmental Hydrology Group

The Environmental Hydrology Group's mission is to provide scientific understanding that leads to the long-term, sustainable management of Australian Rivers, and ensures the effective targeting of catchment and river restoration works. The Group has eleven scientific staff and six support staff.

PROFILES OF STAFF AND TRAINEES

IAEA trainees:

Mr Mubarik Ali, MSc

Senior Geologist, PAEC, Islamabad, Pakistan

Biographical Sketch:

Mr Ali has a MSc in Applied Geology from Punjab University and also a MSc from Q. A. University in Nuclear Engineering in 1988. During the past three years he has been involved in several projects using hydrogen and oxygen isotopic ratios including an IAEA project on using salt affected wasteland for agriculture. He has also determined transit time of lake water using Radon.

Mr Anil Dassanayake, MAgSc

Assistant Soil Chemist, Land Use Division, Department of Irrigation, Colombo, Sri Lanka

Biographical Sketch:

Mr Dassanayake received his MAgSc in Soil Science from University of Reading in 1990. During the past three years he has worked on soil mapping, land suitability for irrigated agriculture, soil erosion hazards and groundwater analysis.

Mr Tommy Hutbarat, MSc

Researcher, CAIR-BATAN, Jakarta, Indonesia

Biographical Sketch:

Mr Hutbarat received his Msc in Physics and Mathematics at the National University, Jakarta in 1991. He has undertaken IAEA training at the Institute of Nuclear Medicine and Oncology, Pakistan in 1990. During the last three years he has worked on projects using ^{210}Pb and ^{137}Cs for dating sediments and measuring sedimentation rates in reservoirs and lakes, heavy metal pollutants and silt as environmental bioindicators and also on the flocculation and settling velocity in sea water.

STAFF Profiles:

Dr Jon Olley, PhD
Research Leader
Environmental Hydrology Group



Biographical Sketch:

Jons' research is aimed at understanding the source and transport of sediment and nutrients in large river systems with a particular focus on understanding how these systems respond to changes in land-use and climate.

Skills and Expertise:

Geology, optical dating, fluvial geomorphology, geochemistry, stable isotope and radioisotope chemistry.

Dr Gary Caitcheon, PhD MSc
(Hons) BSc

Senior Experimental Scientist



Biographical Sketch:

Gary joined CSIRO in 1984 and has mainly worked in the area of sediment tracing, particularly the application of environmental magnetism to sourcing sediment. He has recently become involved in the development of OSL techniques to age date fluvial sediment.

Skills and Expertise:

Fluvial geomorphology and sedimentary geology.

Dr Peter Wallbrink, PhD BSc(Hons)

Research Scientist

Biographical Sketch:

Peter started his research career investigating the response of saturation zones to logging operations near Eden NSW. Since then he has been involved in investigating the fallout and distribution patterns of cosmogenic Beryllium-7, Caesium-137 and Lead-210. He has also been developing techniques to use these radiotracers to measure soil erosion rates and determine sediment sources and their residence times in major rivers of the Murray-Darling Basin. He recently returned to Eden to investigate the influence of forest roads on the water quality of coastal floodplains and investigate the redistribution of sediments occurring within harvested coupes.



Skills and Expertise:

Radionuclide analysis, sediment tracing, soil erosion, impacts of forestry operations, sediment delivery and sediment sources.

Mr Gary Hancock, MSc BSc

Experimental Scientist

Biographical Sketch:

As a specialist environmental chemist Gary has extensive experience studying the behaviour of dissolved phosphorus in rivers. His work also focuses on the measurement and use of environmental radionuclides as tracers of processes (including rates of sedimentation, particle settling, water column mixing, and the movement of solutes across the sediment-water interface) controlling the fate of particulate and dissolved substances in rivers and estuaries.



Skills and Expertise:

Radiochemistry, sediment and water chemistry, and the application of radionuclides to environmental science.

Mr Haralds Alksnis, BA

Senior Technical Officer

Biographical Sketch:

Haralds has 30 years experience with the Division working mainly on projects of a hydrological nature but also on a number of climate studies. During this time he has gained his BA (Mathematics and Geography) from the ANU. Haralds' speciality is in the design and construction of measuring instruments and in managing the gamma ray facility.

Skills and Expertise:

Mathematics and statistics, hydrology, geomorphology, biogeography and gamma technology.



Mr Danny Hunt

Technical Assistant

Biographical Sketch:

Danny joined the Land and Water in 1997 as a contractor working as a field hand with Dr Jacky Croke and the rainfall simulator. He then worked in the Gamma laboratory for 8 months assisting with soil preparation for radionuclide analysis. He is now employed as a technical assistant in the Gamma laboratory, preparing soils for radionuclide analysis, collecting soil samples in the field and generally looking after the technical issues that arise in the Gamma laboratory.

Skills and Expertise:

Member of the Institute of Automotive Engineers of Australia, diesel mechanic, fuel depot fitter mechanic, LP gas fitter, welder, earth moving plant operator, automotive electrical, heavy vehicle driving (4&6 wheel drive), farming, soil preparation.



WORK SCHEDULE

August/September:

- Introduction to CSIRO Land and Water and the gamma spectrometry laboratory of the Environmental Hydrology group.
- Training in preparation of soil and sediment samples for radionuclide analysis
- Training in use of gamma spectrometers to undertake soil and sediment analysis
- Training in production of gamma spectrum from soil and sediment samples
- Training in identifying and analysing major peaks in gamma spectrum from soils and sediments
- training in collation and initial interpretation of gamma data from soils and sediments
- selection of field sites to undertake soil and sediment sampling

October:

- Undertake field work to collect soil and sediment samples
- Preparation of materials collected from field work
- Production of gamma spectrums from field samples
- Preliminary assessment of results from samples from field work
- Training in undertaking Uranium and Thorium analysis by Alpha spectrometry
- Training in electroplate deposition for Radium-226, 228; Thorium-230, 228
- Training in sample preparation for Polonium-210 analysis

November:

- Training in use of conceptual models to interpret sediment depth source
- Training in use of models to interpret spatial source of sediments
- Training in use of mathematical models to determine sedimentation rates
- Application of various models to data collected from field work
- Write summary report on application of models and major findings of research work

METHODS FOR SOIL PREPARATION FOR RADIONUCLIDE ANALYSIS

In this section of the training manual various procedures are outlined that give a broad appreciation for some of the major applications of radionuclide techniques, and some methods for undertaking these applications. These methods take trainees all the way through the process of selecting a field site, preparing samples, analysing them on a gamma spectrometer and obtaining a printout of results. The methods are not meant to be definitive, however they broadly describe the way in which these tasks are undertaken by the Environmental Hydrology group at CSIRO Land and Water. They could well be adapted to suit the different needs, and scientific applications of users in different environments and countries.

The methods of field sampling generally fall into three broad areas:

- Cores taken within sediments deposited on floodplains or within reservoirs - to determine sedimentation rates, spatial origin of material.
- Grab samples of deposited samples taken from within stream lines, or catchment slopes - to determine spatial source of material.
- Cores taken within catchment soils and slopes to determine inventories of fallout radionuclides - to determine erosion rates, source signatures etc.

Each of these is described in more detail in the following pages, beginning with the field methods for obtaining the sample, preparation of that material for subsequent analyses and methods for undertaking analyses of specific tracer properties as required. Following this are sections that deal with 4) Changing samples on detectors; 5) How to obtain a printout of the analysis from each detector and finally 6) Some instructions on how to fill the detectors with liquid nitrogen. A brief overview of the methods utilised for undertaking analysis of radionuclides at the Environmental Hydrology laboratory is given in section 7).

1. ANALYSING DEPOSITED MATERIAL IN RESERVOIRS AND FLOOD PLAINS

Field Methods

A major reason why radionuclide analyses are undertaken on samples is to determine their accumulation rates in reservoirs and floodplains. In this case the first task is to select an appropriate field location for sampling. Sediments from these locations can then be obtained by one of the following methods:

- Vehicle mounted mechanical drilling rig (for accessible floodplain material – good for obtaining cores to depths greater than 2 metres).
- Box section monolith (allows undisturbed sample of stratigraphy to be returned to the laboratory for more complete and detailed analysis – excellent for deposits of less than 2 metres depth).
- Cores using PVC tubing – (quicker than box section monoliths, although surface area representation is generally less – mostly used for deposits < 3 metres depth).
- Boat mounted cores, vibro-hammers, dry ice cores (can all be used to take core samples in various conditions).

Clearly identify the top and bottom of the cores, seal off and pack any free air space at the ends of the cores to prevent movement during transport. Ensure samples are then firmly wrapped and sealed against contamination and loss during transport back to the laboratory. Plastic sheeting, plastic bags and duct tape are suitable materials for this purpose. Any samples put into bags should be clearly marked with waterproof markers in **2 places** to identify samples, and all details entered into a waterproof field notebook.

1.2 Pre-Preparation of core samples for processing in the laboratory

Note: The following are minimum safety procedures that should be undertaken prior to working with soil samples in the laboratory:

- Wear a disposable dust respirator (3M 8170 class P1 or equivalent).
- Wear dustcoat and gloves.
- Wear eye protection (safety glasses).
- Where dust is evident work in front of dust extractor and ensure it is turned on.

The following method applies for Box section monoliths returned to the laboratory:

- Mark the monolith into appropriate depth increments to determine sedimentation rates from top to bottom.
- Divide the monolith in half vertically, one half for analysis, the other half for storage/archiving.

- Remove one half of the monolith in individual depth sections, weigh material from each section; record the mass on the sample preparation form.
- Place each section in suitable containers ready for drying; (mark each container with the section depth and sample number).
- Secure remaining half of monolith in the container box using plastic sheeting and styrofoam, or similar material, to maintain monolith form.
- Securely wrap and seal monolith and store in archive

The following method applies for PVC tube or drill rig cores returned to the laboratory:

- Mark core/tube into appropriate depth increments from top to bottom.
- Slice core into appropriate depth sections.
- Take half of each section for analysis and store the remaining half in plastic bags for archive, mark each bag with sample number and depth.
- Weigh the half to be processed and record mass on the sample preparation form.
- Place sample in a suitable container for drying, and mark each container with the section depth and sample number.

1.3 Drying

Place each batch of samples on trays and place in the circulation oven set at 50°C and allow to thoroughly dry.

When dry, remove samples from circulation oven, weigh immediately and record the dry mass on the sample preparation form.

1.4 Moisture content

Moisture content is calculated using the formula:

$$100 - ((\text{dry mass}) \div (\text{raw mass})) \times 100.$$

1.5 XRF analysis

If XRF analysis is required, take a small amount of each sample to be analysed, and place approximately 1.5g in a vial. Label each vial with sample code and depth.

1.6 Mineral Magnetics

If mineral magnetics analysis is required take a small amount of each sample to be analysed, and place approximately 3.5g in a cuvet. Label each cuvet with sample code and depth.

1.7 Particle size analysis

Sometimes an understanding of the different particle size composition of floodplain, or reservoir sediment is required. The following describes one way in which to undertake a simple particle size analysis of a sample of deposited material. The task is undertaken in two stages, the first involves a physical separation of material down to 63 μm by passage through sieve stacks, the second involves further separation of the remainder material by settling procedures, according to the general principles of Stokes law. Note the detailed method for undertaking settling is given in section 1.8 below.

- Place sample to be sieved into a bowl and re-hydrate with water, allow to stand for approximately 2 days.
- After re-hydrating put sample in a suitable container and place in ultrasonic bath (sonifier) for 10 minutes to help disperse clay/particle aggregates. Refer to the sonifier operator's manual for operating instructions.
- Then place bulk sample into a wet sieve and sieve out particle size fractions as specified on job sheet, e.g. fractions >500 μm , 250-500 μm , 125-250 μm , 63-125 μm , leaving the <63 μm fraction in a bucket.
- The <63 μm fraction is then settled to the particle size fractions required on job sheet e.g.,
 - 40-63 μm ,
 - 20-40 μm ,
 - 10-20 μm ,
 - 2-10 μm
 - <2 μm
- Use a settling cylinder or bucket depending on the quantity of sample to be settled, (refer to settling procedure).
- Once all size fractions have been sieved/settled out they are then placed in bowls or buckets and dried in the circulation oven.
- When dry, weigh each size fraction and record the mass in the sample preparation form.
- Place fractions in separate bags and identify each bag for further processing or storage.

Note: At all stages of sieving and settling, buckets and bowls used must be clearly identified with sample code and size fraction.

1.8 Settling Procedure

Prepare settling cylinder:

- Place a mark 5cm from the bottom of the cylinder, this allows for the volume remaining in the cylinder after extraction to be equal in proportion with a series of settling cylinders.
- Place a mark 30cm from the bottom of the cylinder, this mark allows for a fall depth of 25cm.

Settling:

- Place the <63µm fraction in the settling cylinder and top up with water to the 30cm mark.
- Mount siphons in the cylinder with lower edge of the tube set at 5cm from the bottom of the cylinder i.e. at the 5cm mark.
- Mix the sample thoroughly in the cylinder and allow to stand (settle) for the following times:

Time	Fraction settled	Fraction in suspension
3 mins	40 – 63 microns	<40 microns
12 mins	20 – 40 microns	<20 microns
48 mins	10 – 20 microns	<10 microns
20 hrs	2 – 10 microns	<2 microns

- Siphon off suspended fraction after the 3 min settling time for the 40-63 µm particle size fraction.
- Replenish water to the 30cm mark and repeat the process for the remaining size fractions until the desired particle size fractions have been removed.

Note: - These times are used when settling is performed at a constant temperature of 20°C, for temperature correction times refer to chart in appendix A.

- Place the settled size fractions in the circulation oven at 50°C and dry.
- When dry, weigh each size fraction and record the mass in the sample preparation form.
- Place fractions in separate bags and identify each bag for further processing or storage.

2. DEPOSITED SEDIMENTS FROM CHANNELS AND SLOPES

2.1 Field Methods

The tracer properties of deposited material in channels can be analysed to determine its spatial origin as well as the erosion process responsible for its detachment and transport. After consideration of the experimental design, select the field site appropriate and collect samples in plastic bags. Collection should be undertaken along representative reaches of river, and represent as many small replicates of material as possible. In this way the sample will be 'averaged', thus representing the characteristics of the sediment deposits as best as possible.

Collect approximately 1 to 1.5 kg of raw material, depending on type and density of the soils. Heavy clays require approximately 1kg, whereas coarse aggregates may require a greater mass to produce a sufficient quantity of various particle sizes for casting.

Clearly mark bags in **2 places** to identify samples, and enter sample code details into field notebook. Samples are taken in the various field sites using a small spade or scoop. Seal plastic bags with elastic bands.

2.2 Pre-Preparation of deposited sediment samples

Weigh each sample and record the mass in the sample preparation form.

For a wet sample:

- Place sample in a bowl and thoroughly mix.
- Select a suitable plastic container (49x85 securitainer vial), weigh the container and record the mass.
- Take sub-sample approximately 10% of total mass and record the sub-sample mass in the sample preparation form.
- Dry the sub-sample in the circulation oven and record the dry mass in the sample preparation form.
- Store sub-sample in archive

For a dry Sample

- Thoroughly mix sample in the bag and empty onto clean dry surface.
- Select a suitable plastic container (49x85 securitainer vial or similar), weigh the container and record the mass.
- Smooth out sample to approximately 2 cm thick and divide the sample into 10cm grid squares.
- Take a small amount of sample from each grid and place in sub-sample container, total sub sample approximately 10% of total mass.

- Dry the sub-sample in the circulation oven and record the dry mass in the sample preparation form.
- Store sub sample in the archive.

After sub-samples are taken, place bagged samples on trays and place in circulation oven at 50°C and allow to thoroughly dry.

When dry, remove samples from oven, weigh immediately and record the dry mass in the sample preparation form.

If necessary the following analysis may be required.

2.3 Moisture content

Moisture content is calculated using the formula:

$$100 - ((\text{dry mass}) \div (\text{raw mass})) \times 100.$$

2.4 XRF analysis

If XRF analysis is required, take a small amount of each sample to be analysed, and place approximately 1.5g in a vial. Label each vial with sample code and depth.

2.5 Mineral Magnetism

If mineral magnetism analysis is required take a small amount of each sample to be analysed, and place approximately 3.5g in a cuvet. Label each cuvet with sample code and depth

2.6 Particle size analysis

Carry out particle size analysis as described on section 1.7, page 12.

3. DETERMINING INVENTORIES OF FALLOUT RADIONUCLIDES FROM SOIL CORES

3.1 Field methods

This section describes methods for taking samples to determine the inventories of fallout radionuclides in soils. This information is often used to determine 'reference' inventory amounts as well as the difference between these sites and those that have undergone erosion and deposition. Firstly the experimental design should be carefully considered, and the appropriate field site selected. Important factors here are the inherent spatial variability of these fallout nuclides, as well the long-term (40 year) history of erosion and deposition at the study areas.

3.2 Collecting sample cores

- Take the cores in either a transect (straight line) or in a grid pattern. A minimum of five samples is generally taken. If a transect is used, the core positions must be evenly spaced (at least approximately 30m-50m apart), if a grid is used minimum spacings of 20-30m should also be considered.
- The cores are taken using 75 mm hand augers and are taken at depths up to 30cm, in increments of progressively increasing depth i.e., 0-2cm, 2-5cm, 5-10cm, 10-15cm, 15-20cm, 20-25cm, 25-30cm.
- If combining samples from an area to obtain a single mean value, then the samples from each depth are collected, weighed individually on field scales, then put into a bag and mixed together. For example the 0-2cm sample from one core position is weighed individually and then put into a bag with the weighed 0-2cm increment from the next core etc. until all the samples at the 0-2cm depth are collected into 1 bag.
- This is then repeated for the next depth increment (2-5cm) sample from each core position, which is also weighed individually and put into the next bag until all samples at the 2-5cm depth are collected into 1 bag.
- This method is repeated until an average sample from each depth increment is collected from all core positions.
- Clearly mark all bags in **2 places** to identify samples, and enter sample code details and locations into field notebook.

3.3 Soil preparation for analysing ^{137}Cs and ^{210}Pb inventories from soil cores

- Weigh each combined sample and record the mass in the sample preparation form.
- Place bagged samples on a tray and dry in the circulation oven set at 50°C .
- When samples are thoroughly dried, remove them from the circulation oven.
- Weigh each sample and record the mass in the sample preparation form.
- Thoroughly mix sample in a bowl and empty onto clean dry surface.
- Select a suitable plastic container (49x85 securitainer vial or similar), weigh the container and record the mass.

- Smooth out sample to approximately 2 cm thick and divide the sample into 10cm grid squares.
- Take a small amount of sample from each grid and place in sub-sample container, total sub-sample approximately 10% of total mass.
- Record the sub-sample mass on the sample preparation form.
- Store sub-sample in the archive.
- Take a representative sample from each grid square and put into foil containers ready for ashing prior to casting.
- The total mass of each sample required for ashing is approximately 300g.

3.4 Ashing of soil samples prior to casting for radionuclide analysis

Soil samples for radionuclide analysis are ashed in an oven set at 450⁰C to remove any organic material that may be present.

Then:

- Scribe sample codes and depths onto foil containers.
- Place sample to be analysed in foil containers ready for ashing, (approximately 280grams for cups, 60 grams for discs, 15 grams for sticks)

Note: Cast geometry depends on the mass of sample available for casting.
Desired ashed sample mass is:

- Cup 250 grams
- Disk 40 grams
- Stick 10 grams

- Weigh pre ashed sample and record the mass in the sample preparation form.
- Close foil containers to prevent cross contamination of samples during ashing.
- Place samples in oven set at 450⁰C and ash for 2.5 days or until completely ashed.
- Remove ashed sample from oven and allow it to cool, re weigh sample as soon as it is cool and record the mass in the sample preparation form.
- Calculate and record the loss on ignition percentage using the formula :

$$((\text{pre-ashed mass}) - (\text{ashed mass})) \div ((\text{pre-ashed mass}) \times 100)$$

- Grind the sample to a uniform consistency in the large soil grinder, (soil container, lid and puck must be thoroughly cleaned between each sample).
- Soil sample is then ready to be cast into the various geometries depending on mass of sample available.

3.5 Casting of ashed sample for radionuclide analysis

After ashing soil samples are cast into the appropriate geometries using, polyester rapid cure clear resin and MEKP (methyl ethyl ketone peroxide) hardener. The following safety procedures are considered essential prior to casting:

- Read safety instructions on resin and hardener containers prior to use.
- Wear dustcoat and disposable vinyl gloves.
- Wear safety glasses.
- Wear half mask respirator with class A1 filter.
- Ensure casting room ventilation fans are switched on and working.

Prior to casting samples, the aluminium moulds must be thoroughly cleaned and coated with PLP10 paste wax release agent.

Identify moulds with cast code using a marker pen.

For mixing bowls use polypropylene food bowls of a suitable size to suit the geometry being cast.

- Fill in radioisotope sample preparation form recording :
 - Cast date
 - Cast code
 - Sample code
 - Time
 - Your initials
 - Project/sample details
- Weigh and record the mixing container and mixing rod mass.
- Re zero the balance, weigh and record the total sample mass to be cast.
- Record the quantity of hardener used (4 ml for cups, 2 ml for discs, 2 drops for sticks).
- Add resin to obtain a stiff but workable mixture, thoroughly mix sample, resin and hardener. Mixture must be of a stiff consistency; otherwise resin and sample segregation may occur, resulting in inaccurate analysis results.
- Record total mixture mass.

Total mixture mass depends on soil density, an approximate guide is:

- Cup 380 grams
 - Disk 60 grams
 - Stick 12 grams
-
- Pour sample mixture into the mould starting from one edge, (this may require assistance with the mixing rod) then tap mould on top of bench to settle mixture to bottom of mould.
 - When mould is full place lid on top and seat into position (some mixture loss through holes in lid may occur).
 - Place moulds in fume cupboard and lower the doors, ensure fan is running.
 - Allow mixture to cure (normally 2-3 days).

- Once cured, scribe cast code on top of cast with an electric etching tool.
- Remove cast from mould using nylon hammer if necessary.
- De-burr cast, then weigh and record final cast mass in radioisotope sample preparation form.
- Calculate the net sample in cast using the formula:

$$(\text{total sample mass}) \times (\text{mass of final cast}) \div (\text{total mixture mass})$$

- Record result in radioisotope sample preparation form.
- Thoroughly clean the moulds using steel wool and hot water, and return them to the storage shelf.

3.6 Identification system for samples cast in polyester

Each sample carries its own sample code, plus unique identification code. These are written onto the polyester resin cast itself, as well as an accompanying card. This latter card contains all the relevant details of the sample and its cast. This card follows the sample through the analytical process, and is used to record all the detector information necessary to analyze the sample when it has been counted on the gamma spectrometers. Thus a colour-coded card for each cast needs to be written up including identification and sample mass information. The colour is determined by the colour of the cards stored in the alpha numeric filing system in the detector room. Information required on the cards is:

- Cast date
- Cast code
- Sample number
- Sample details
- Mass of total sample cast
- Final cast mass
- Sample in cast mass

The details for completion of the cards are taken from the Radioisotope Sample Preparation form.

The casts are now placed in storage shelves and ^{210}Pb allowed to reach equilibrium for 23 days prior to radionuclide analysis.

If Beryllium analysis is required the 23 day wait is waived and analysis is commenced as soon as possible.

When the samples have waited the appropriate time they are now ready for analysis on the gamma spectrometers. This process is outlined in section 4) below.

4. CHANGING SAMPLES ON DETECTORS

As of September 1999 there are 5 detectors in use at CSIRO Land and Water, these are referred to as detectors A, B, C, D, & G. Detectors A, B, C, D are operated from MCA1 board. Detector G is operated from MCA2 board.

Written below is a simple step by step procedure to enable trainees to change samples on the detectors, place elapsed time, detector code, storage disk and date and time information in the log books, and to stop and restart detectors to acquire new spectra.

- Write sample information into log books
i.e. Date Time Cast code (eg SD164B.01) and Sample description.
- Write cast code, sample code, and date on the back of sample information cards.
- Take new samples and sample information cards into detector room and place on top of appropriate detectors.
- Remove old sample information cards from detectors and bring into laboratory.
- Observe `C:\SAMDAT>` on computer screen.
- Enter change command (write file names backwards)
e.g. `CHANGE1 A761BS.01 B042DW.01 C881BX.01 D920BG.01`
`CHANGE2 G950JX.01 BLANK BLANK BLANK`
- Press return after checking file names and sufficient disk space (64K of disk space is required to save the spectrum).
- Write count time (ksec) and disk number on the sample information cards and in log books.
- Clear spectra (OK - Ctrl E)
- Proceed to detector room and put new samples on detectors.
- Place old samples in storage crates for archive
- Start detectors counting by pressing F4.
- Leave the system (press alt F4 – NO – Alt F4 – OK)
- Check saved file names with cards

If a mistake is made use the command CONTROL C to abort, and start again.

5. OBTAINING A PRINT OUT OF THE ANALYSIS

- In order to obtain a hardcopy printout of the gamma spectrometer analysis, enter the command EDIT HCOM* A,B,C,D or G, on the computer. Note: Use one letter only, depending which detector analysis is being printed, e.g. for samples analyzed on detector A used EDIT HCOMA.

Note: EDIT HCOM* - Normal samples
ZCOM* - Standards
YCOM* - Backgrounds

- Press insert key
- Update sample information on screen. File names, sample info, dates, weights and times.
- Leave edit file (ESC E).
- Type HUNCH then enter
- Type HCOM* (A, B, C, D, or G) then enter.
- If correct peaks are found press enter, if not, press 1 then type in correct numbers.
- Type FACT and enter, then type of cast and detector (screen will prompt with description and number).
- Type in file name e.g. WD269C.01
- Type PRINTT WD269C.01
- Check print-out.

6. FILLING LIQUID NITROGEN DEWARS

Liquid Nitrogen Dewars need to be filled on all operational detectors.

NOTE: Remember liquid nitrogen is stored at approximately -196°C and can cause severe burns if it comes into contact with any part of your body. It is absolutely necessary for your personal protection that the following steps are taken:

- Wear eye protection
- Wear gloves
- Door to the detector room must be open for your protection (proper ventilation) and in order to operate the micro switch for the solenoid.

Before filling the detectors with liquid nitrogen the computer MCA acquiring system must first be stopped. The steps required to stop the MCA system are as follows:

Enter `CD(space)\SAMDAT` on the computer (use this command only if SAMDAT is not on screen).

At the `C:\SAMDAT>` prompt:

- Type **G** and press **enter**
- MCA 1 hi-lighted click on **OK**
- Press **F4** (start/stop) and check that green light goes off in box **[Full:VFS=4096]**
- Press **Alt F4** click on **NO** to save current spectrum
- MCA.EXE to get to MCA 2
- When MCA.EXE is selected click on **OK**
- Press **F4** to stop counting.

Proceed to detector room and switch on pump and purge liquid nitrogen fill tube by closing outside vent valve and opening fill valve (wait for liquid nitrogen to appear at end of delivery tube) then proceed to fill each Dewar in turn. Note there will be lots of gas venting into the room while the liquid nitrogen line cools down to operational temperature, this represents condensation from atmosphere. When filling is complete, close door a fraction to shut off solenoid; close fill valve and open outside vent valve. For more details see Appendix A.

When all detector Dewars have been filled:

- Press **F4** to turn on green light **[Full:VFS=4096]**
- Press **Alt F4** - Click **NO** to save or not
- MCA.EXE
- MCA 1 Press **OK**
- Press **F4** to turn green light on **[Full:VFS=4096]**
- Press **Alt F4**
- Click **NO** to save current spectrum
- Press **Alt F4** to cancel windows

7. GAMMA LABORATORY METHODS

The Gamma Laboratory has five older gamma detectors and four new detectors as well as eight alpha detectors. Three new detectors have very large Germanium crystals of 80mm diameter by 25mm thickness. One detector is shown at right with a 65mm diameter sample located in a perspex centring ring. For a given sample mass such large detectors show improved efficiency for thin samples.

Other detectors can handle different sample geometries, eg. well detectors are used for small samples that are cast into small cylinders called 'sticks'.



7.1 Calibration

The gamma detectors are calibrated relatively using standard sources of known activity. This eliminates problems in determining efficiency and with true coincidence summing. The sources are diluted with sand, ground and cast in polyester resin.

7.2 Backgrounds

The detectors are shielded from external background radiation by 100mm of lead with a 10mm inner steel box to attenuate lead X-rays. The residual background is measured by acquiring a spectrum with a resin only disk monthly and subtracting the various peak values from each sample peak.

7.3 Counting

Spectra are accumulated for ~84ksec except for well detectors where ~178ksec is accumulated. As well as backgrounds a ~400Bq/kg U/Th standard and a low-activity soil 'standard' are counted monthly to check on detector stability.

7.4 Errors

Errors are linearly related to sample mass so doubling the mass halves the error. The square root of count time is related to the error so 100 days is needed to reduce the error to one tenth of the one day error. Generally every effort to obtain the maximum sample mass of the selected geometry is worthwhile.

8. ACKNOWLEDGMENTS

The authors would like to thank Frances Marston and Alan Marks for assistance with the staff profiles and pictures and Jackie Wraight for coordinating the IAEA trainees visit.